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BEFORE THE BOARD OF PATENT APPEALS
AND INTERFERENCES

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*Ex parte* DANIEL T. COLBERT, HONGJIE DAI, JASON H. HAFNER, ANDREW G. RINZLER, RICHARD E. SMALLEY, JIE LIU, KENNETH A. SMITH, TING GUO, PAVEL NIKOLAEV, and ANDREAS THESS

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Appeal 2008-3765
Application 10/670,955
Technology Center 1700

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Decided: September 30, 2008

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Before CHUNG K. PAK, TERRY J. OWENS, and ROMULO H. DELMENDO, *Administrative Patent Judges*.

DELMENDO, *Administrative Patent Judge.*

DECISION ON APPEAL

STATEMENT OF THE CASE

Appellants appeal under 35 U.S.C. § 134(a) from a final rejection of all pending claims 84, 85, 91, 92, 94, and 95. (Appeal Brief filed July 5,

2007, hereinafter "App. Br."; Final Office Action entered September 11, 2006). We have jurisdiction under 35 U.S.C. § 6(b).

We AFFIRM.

Appellants' invention relates to a method for producing end-derivatized carbon nanotubes. According to Appellants, "tubular carbon molecules (including the multiwall forms) . . . can be chemically derivatized at their ends (which may be made either open or closed with a hemi-fullerene dome)." (Spec. at 34, ll. 11-13). Appellants state:

> Derivatization at the fullerene cap structures is facilitated by the well-known reactivity of these structures. . . . Alternatively, the fullerene caps of the single-walled nanotubes may be removed at one or both ends of the tubes by short exposure to oxidizing conditions . . . and the resulting open tube ends maybe [sic] derivatized using known reaction schemes for the reactive sites at the graphene sheet edge.

(*Id.*, ll. 13-15).

Representative claim 84 reads as follows:

> 84. A method for producing end-derivatized single-wall carbon nanotubes comprising the steps of:
>
> a) providing a plurality of single-wall carbon nanotubes; and
>
> b) reacting the single-wall carbon nanotubes with a compound that provides at least one substituent on at least one of the ends of at least a portion of the single-wall carbon nanotubes.

The prior art references relied upon by the Examiner to reject the claims on appeal are:

| | | |
|---|---|---|
| Hiura | 5,698,175 | Dec. 16, 1997 |

Sumio Iijima and Toshinari Ichihashi, *Single-Shell Carbon Nanotubes of 1-Nm Diameter*, 363 NATURE 603-605 (1993) (hereinafter "Iijima").

The following rejection[1] is before us for review:

Claims 84, 85, 91, 92, 94, and 95 are rejected under 35 U.S.C. § 103(a) as unpatentable in view of the combined teachings of Hiura and Iijima.

## ISSUE

Have Appellants shown reversible error in the Examiner's determination that the subject matter of claims 84, 85, 91, 92, 94, and 95 would have been obvious to one of ordinary skill in the art in view of the combined teachings of Hiura and Iijima?

## FINDINGS OF FACT

The record supports the following findings of fact, as well as any other findings of fact discussed in this opinion, by at least a preponderance of the evidence.

1. Iijima discloses "the synthesis of abundant single-shell [carbon nano]tubes." (Iijima at 603, 1st paragraph).
2. Hiura describes "a process for purifying, uncapping and chemically modifying carbon nanotubes." (Col. 1, ll. 6-7).
3. Hiura discloses: "[A] process for obtaining carbon nanotubes to which various functional groups such as nitro-, sulfon-, carboxyl-[,] carbonyl-, ether-[,] or phenolic hydroxyl- group and the like are introduced." (Col. 3, ll. 30-33).

[1] In the Examiner's Answer, the Examiner states that the rejection of claims 94 and 95 under 35 U.S.C. § 112, ¶1, is withdrawn. (Ans. 2).

4. Hiura does not expressly limit the wall type (i.e., multi-wall or single-wall) of nanotubes used with the disclosed liquid phase process for producing end-derivatized carbon nanotubes.
5. Hiura describes the "conventional process for purifying and uncapping carbon nanotubes is conducted in a manner that they are oxidized under an atmosphere with an oxidative gas such as oxygen, steam or the like at a high temperature." (Col. 1, ll. 22-25).
6. Hiura explains that the high reaction temperature of the "conventional process for purifying and uncapping carbon nanotubes" makes it "difficult to control or select an optimum time and temperature for the reaction." (Col. 1, ll. 34-38).
7. Hiura discloses a liquid phase process where "purified nanotubes which have a certain quality can be easily obtained at high yield by milder condition [than the conventional process] since the reaction is proceeded in liquid phase." (Col. 2, ll. 50-56).
8. Hiura discloses "the process of the invention has higher selectivity than the conventional process." (Col. 5, ll. 2-3).
9. Hiura teaches: "[I]t is found that the erosion of a nanotube, i.e., the problem of the conventional process by which the nanotube shortens rapidly during oxidation proceeds is not observed." (Col. 5, ll. 4-6).
10. Hiura discloses that purifying nanotubes with the milder reaction condition of the liquid phase process allows control of the degree of reaction, and "if the time of reaction is controlled, then nanotubes which have various states of proceeding of reaction may be obtained." (Col. 6, l. 64 through 7, l. 1).
11. Hiura's Figure 3 is reproduced below:

FIG. 3

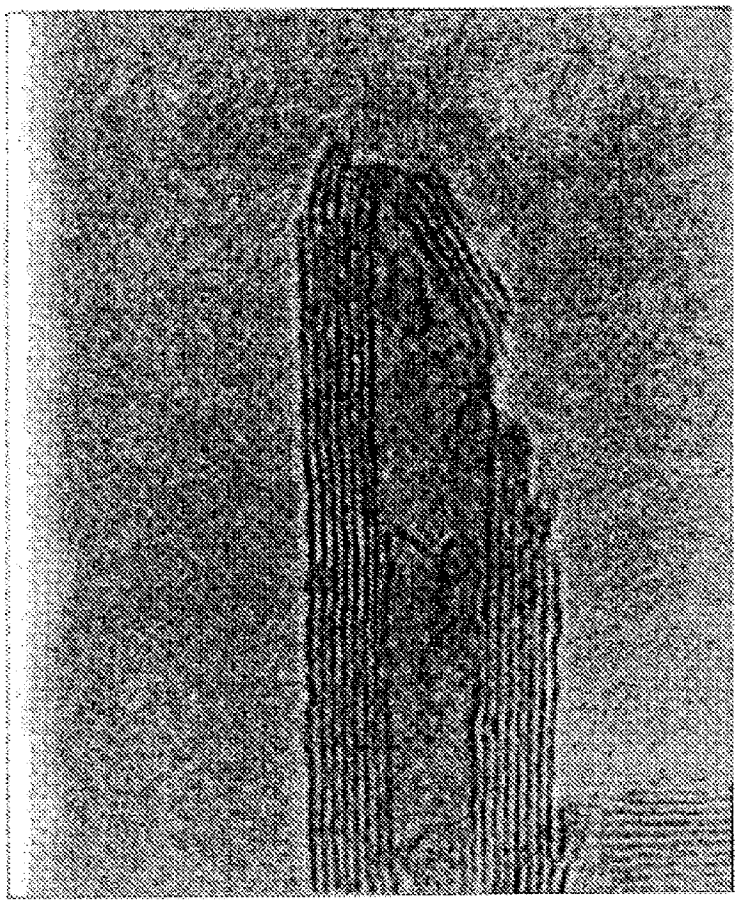

Figure 3 depicts a magnified TEM (Transmission Electron Microscopy) image of an uncapped nanotube after treatment with a mixture solution of sulfuric acid and nitric acid. (Col. 3, ll. 65-67; col. 4, l. 66 through 5, l. 2).

12. Dujardin[2] states:

> It was believed that purification of single-shell nanotubes based on preferential oxidation and/or separation using surfactants was much more difficult than that of

[2] Erik Dujardin et al., "*Purification of Single-Shell Nanotubes*," 10 ADV. MATER. 611-613 (1998) (App. Br. Exhibit A) (hereinafter "Dujardin").

> multishell nanotubes. For instance, gas-phase oxidation, which yields purified multishell nanotubes, destroys the single-shell nanotubes before anything else in the sample.

(Endnotes omitted; Dujardin at 611, 2nd paragraph).

13. Dujardin refers to 1995 Hiura[3] at the end of his statement that "[i]t was believed that purification of single-shell nanotubes based on preferential oxidation and/or separation using surfactants was much more difficult than that of multishell nanotubes," not in reference to his statement that gas-phase oxidation destroys single-shell nanotubes. (*Id.*).

14. Dujardin was published after the publication dates of Hiura and Iijima, as well as after Appellants' effective filing date (acknowledged by the Examiner and Appellants). (Ans. 5, l. 20 through 6, l. 2; App. Br. 8, ll. 24-27; Reply Br. 8, ll. 8-10).

15. Thomas W. Ebbesen, one of the authors of Dujardin, is also a co-inventor of Hiura. (Dujardin at 611).

## PRINCIPLES OF LAW

The question of obviousness is resolved on the basis of underlying factual determinations including: (1) the scope and content of the prior art; (2) the level of ordinary skill in the art; (3) the differences between the claimed invention and the prior art; and (4) secondary considerations of

[3] Hidefumi Hiura et al., "*Opening and Purification of Carbon Nanotubes in High Yields*," 7 ADV. MATER. 275-276 (1995) (App. Br. Exhibit B) (hereinafter "1995 Hiura").

nonobviousness, if any. *Graham v. John Deere Co.*, 383 U.S. 1, 17-18 (1966).

"Obviousness [under 35 U.S.C. § 103] does not require absolute predictability of success . . . all that is required is a reasonable expectation of success." *In re O'Farrell*, 853 F.2d 894, 903-904 (Fed. Cir. 1988) (citations omitted).

"[I]n considering the disclosure of a reference, it is proper to take into account not only specific teachings of the reference but also the inferences which one skilled in the art would reasonably be expected to draw therefrom." *In re Preda*, 401 F.2d 825, 826 (CCPA 1968).

Design need or market pressure to solve a problem from a finite number of identified, predictable solutions provides good reason for a person of ordinary skill in the art "to pursue the known options within his or her technical grasp. If this leads to the anticipated success, it is likely the product not of innovation but of ordinary skill and common sense." *KSR Int'l Co. v. Teleflex, Inc.*, 127 S. Ct. 1727, 1742 (2007).

"Attorney's argument in a brief cannot take the place of evidence." *In re Pearson*, 494 F.2d 1399, 1405 (CCPA 1974).

## ANALYSIS

Appellants argue claims 84, 85, 91, 92, 94, and 95 together. Accordingly, we select claim 84 as representative and confine our discussion to this claim. 37 C.F.R. § 41.37(c)(1)(vii).

Appellants do not contest the Examiner's finding that Hiura teaches a method for end-derivatizing carbon nanotubes comprising reacting carbon nanotubes to provide at least one substituent on at least one of the ends of at

least a portion of the carbon nanotubes. (Ans. 3, ll. 11-15; App. Br. 5-13; Reply Br. 2-11; FF 2 and 3). Rather, Appellants argue that it would not have been obvious to employ the processes disclosed in Hiura to produce end-derivatized *single-wall* carbon nanotubes. In particular, Appellants contend that the processes disclosed in Hiura relate to *multi-wall* carbon nanotubes and there are "structural differences between single-wall and multi-wall carbon nanotubes [that] lead[] to differences in physical and chemical properties, such as tensile strength, modulus, flexibility, thermal conductivity, electrical conductivity, chemical reactivity and chemical stability." (App. Br. 7, ll. 26-29). Appellants assert:

> Single-wall carbon nanotubes are molecules of carbon, while multi-wall carbon nanotubes are assemblies of carbon. Single-wall carbon nanotubes have only a single layer of $sp^2$-hybridized carbon atoms generally arranged in hexagons and pentagons. Because of their single-layer, single-wall carbon nanotubes generally cannot support defects in growth and are more susceptible to destruction by bond breakage or reaction. In contrast, multi-wall carbon nanotubes are composed of multiple, cylindrical concentric carbon layers arranged in a nested or scrolled fashion. Because of this arrangement, the carbon shells of multi-wall carbon nanotubes can withstand wall defects, which often appear as dislocations, kinks, holes, edges on the side-wall surfaces, *etc*. Also because of their multiple layers and the interconnections between these layers, multi-wall nanotubes (in comparison to single-wall nanotubes) can withstand much more rigorous chemical processing, physical conditions, and extensive chemical bond breakage without nanotube destruction.
>
> Single-wall carbons nanotubes "rope" together and are held tightly by van der Waals forces. As such, single-wall nanotubes are difficult to separate and disperse in other media, while multi-wall nanotubes do not share the same propensity to rope and, as such, are readily separable and dispersible.

(*Id.* 7, ll. 12-26). Appellants assert that these differences result in "chemistry that can be done with each species [that] is quite different and would be unpredictable." (*Id.* 8, ll. 1-2).

Appellants' arguments are unpersuasive. Iijima discloses "the synthesis of abundant single-shell [carbon nano]tubes." (FF 1). Hiura describes "a process for purifying, uncapping, and chemically modifying carbon nanotubes." (FF 2). In particular, Hiura discloses producing end-derivatized carbon nanotubes with "a process for obtaining carbon nanotubes to which various functional groups such as nitro-, sulfon-, carboxyl-[,] carbonyl-, ether- or phenolic hydroxyl- group and the like are introduced." (FF 3). While not expressly disclosing single-wall nanotubes, Hiura places no limitations on the wall type of nanotubes used with the disclosed liquid phase process for producing end-derivatized carbon nanotubes. (FF 4). Accordingly, Hiura suggests that all known nanotubes, including single-wall nanotubes as shown in Iijima, would work in the disclosed process of chemically modifying carbon nanotubes to produce end-derivatized carbon nanotubes. Thus, one of ordinary skill in the art would have found obvious to combine the teachings of Iijima and Hiura to obtain a method of producing end-derivatized single-wall carbon nanotubes. *O'Farrell*, 853 F.2d at 903-904. *Cf. In re Preda*, 401 F.2d at 826 ("[I]n considering the disclosure of a reference, it is proper to take into account not only specific teachings of the reference but also the inferences which one skilled in the art would reasonably be expected to draw therefrom.").

Appellants' assertions of differences between multi-wall nanotubes and single-wall nanotubes are not persuasive evidence of unpredictability or lack of expectation of success. These assertions are unsubstantiated by any

data or other evidence to show that the chemical reactivity of single-walled nanotubes is unpredictable. Moreover, Appellants do not submit any evidence comparing properties between end-derivatized single-wall nanotubes and end-derivatized multi-wall nanotubes to demonstrate unexpected results. (App. Br. 10, ll. 26-30). *Pearson*, 494 F.2d at 1405 ("Attorney's argument in a brief cannot take the place of evidence.").

Appellants argue that "the teachings of Hiura when combined with the teachings of *Iijima* [would] not have suggested a reasonable likelihood of success as applied to single-wall carbon nanotubes, such a chemical protocol [disclosed by Hiura] would be expected to fail to achieve the desired results." (App. Br. 8, ll. 15-17). Appellants argue that "multi-wall carbon nanotubes, typically riddled with defects, results in the breaking of carbon-carbon bonds generally at regions of defects on the walls as well as the ends." (*Id.* 8, ll. 8-9). Appellants contend Hiura's Fig. 3, showing damage occurring to multi-wall nanotubes by Hiura's process, would lead one of ordinary skill in the art "to believe such processes would destroy" single-wall nanotubes. (*Id.* 8, l. 14).

Appellants' arguments are unpersuasive. Hiura's Figure 3 shows the uncapped nanotubes after treatment with a mixture solution of sulfuric acid and nitric acid. (FF 6, 12; col. 4, l. 25 through 5, l. 2). Hiura's nanotube wall structure shown in Fig. 3 is not persuasive evidence that the Hiura process would inevitably or necessarily destroy single-wall nanotubes. Rather, it would appear that similar breakage of carbon-carbon bonds in the wall structure of single-wall nanotubes would merely result in shorter single-wall nanotubes. Furthermore, Hiura discloses that the liquid phase process of producing end-derivatized carbon nanotubes is a controllable reaction,

that the "problem of the conventional process by which the nanotube shortens rapidly during oxidation proceeds is not observed, and that "nanotubes which have various states of proceeding of reaction may be obtained." (FF 7, 9, 10). Thus, one of ordinary skill in the art would have understood that, with Hiura's controlled reaction of the degree of purification and uncapping, there would have been an anticipation of success in obtaining end-derivatized single-wall nanotubes, even if the single-wall nanotubes cannot withstand as rigorous chemical processing as multi-wall nanotubes, as asserted by Appellants. *KSR*, 127 S. Ct. at 1742. Moreover, Appellants have not shown that their claimed method is free from similar carbon-carbon bond breakage.

Appellants have not directed us to any persuasive evidence that Hiura gives any reason to believe that the disclosed process of producing end-derivatized nanotubes would destroy single-wall nanotubes. Accordingly, Appellants have failed to show Examiner error in determining the claimed subject matter obvious in view of Hiura and Iijima.

Appellants argue that Dujardin, published after the publication dates of Hiura and Iijima, as well as after Appellants' effective filing date (FF 14), expresses the view that "the Hiura process would destroy the single-wall carbon nanotubes." (App. Br. 8, 22-27). Therefore, Appellants contend that "one of ordinary skill in the art would have been discouraged to apply the process of *Hiura* to the nanotubes of *Iijima* because there would not be a reasonable expectation of success." (*Id.* 9, ll. 13-15). Particularly, Appellants assert:

> *Dujardin* notes that "[i]t was believed that purification of single-shell nanotubes based on preferential oxidation and/or separation using surfactants was much more difficult that [sic]

> that of [multi-shell] nanotubes." *Dujardin*, at 611. A reason for this was because "gas-phase oxidation, which yields purified multishell nanotubes, destroys the single-shell nanotubes before anything else in the sample.

(FF 12; App. Br. 8, l. 28 through 9, l. 2). Also, Appellants assert that because one of the authors of Dujardin (i.e., Ebbesen) is also a co-inventor of Hiura (FF 15) "there can be no dispute that the authors of *Dujardin* were fully cognizant of the processes disclosed in *Hiura*, when providing this statement." (*Id.* 9, ll. 24-27). Therefore, Appellants contend: "[T]he authors of Dujardin expressly stated that oxidation processes (like those disclosed in Hiura) would work on multi-wall carbon nanotubes, but would destroy single-wall carbon nanotubes before anything else." (Reply Br. 4, ll. 25-27).

We find Appellants' arguments unpersuasive. Appellants have not established that the difficulty in purifying single-wall nanotubes, as stated in Dujardin, was due to total destruction of the single-wall nanotubes and not, for example, merely due to low yields. Also, Appellants have not persuasively established Dujardin's statement regarding the gas-phase oxidation, which destroys single-wall nanotubes, refers to Hiura's liquid phase process. Furthermore, Appellants have not established Dujardin's statement of what "was believed" was actually accepted knowledge at the time of the invention, and not just a past belief that was no longer accepted by one of ordinary skill in the art at the time of Appellants' invention.

Appellants contend "the statement made in *Dujardin* applies to liquid-phase oxidation (as well as gas-phase oxidation) of multi-shell nanotubes." (App. Br. 10, ll. 7-8). In particular, Appellants contend that Dujardin's statement references to four papers at endnotes 5-8 (App. Br. 9, l. 28 through

10 l. 2), and that endnote 6 (1995 Hiura) describes "opening and purifying multi-walled carbon nanotubes" with "oxidants 'such as nitric acid, sulfuric acid, [and] the mixture of both and potassium permanganate.'" (*Id.* 10, ll. 5-6). Appellants argue that this disclosure shows that "the statement made in *Dujardin* applies to liquid-phase oxidation (as well as gas-phase oxidation) of multi-shell nanotubes," and thus, "a person of ordinary skill in the art would not have expected, at the time of the invention, the results shown in the present Application." (*Id.*, 10, ll. 7-10).

Appellants' arguments are unpersuasive. Significantly, 1995 Hiura is cited in Dujardin after the statement concerning the relative difficulty in purifying single-shell nanotubes as compared to multi-shell nanotubes, not in reference to the gas-phase destruction of single-shell nanotubes. (FF 13). Furthermore, Appellants do not point to any evidence in 1995 Hiura to show that one of ordinary skill in the art would have understood single-wall nanotubes to be destroyed in the Hiura liquid phase process.

Moreover, Appellants' arguments directed to a specific type of oxidation purification process (i.e., gas-phase) is not persuasive evidence that one of ordinary skill in the art, at the time of the invention, would have been led to believe the claimed derivatization process had no expectation of success. Though Dujardin relates the prior art gas-phase oxidation to destruction of single-wall nanotubes, Hiura discloses a controlled liquid phase process to produce end-derivatized nanotubes, which is milder, more selective, and more controllable than the conventional gas phase reaction. (FF 5-10).

For these reasons, we find Appellants have not shown that the Examiner erred in determining claims 84, 85, 91, 92, 94, and 95 obvious over the combined teachings of Hiura and Iijima.

## CONCLUSION

Appellants have failed to show that the Examiner reversibly erred in concluding that one of ordinary skill in the art would have found the subject matter of appealed claims 84, 85, 91, 92, 94, and 95 obvious in view of the combined teachings of Hiura and Iijima.

Accordingly, the decision of the Examiner to reject all the appealed claims is affirmed.

No time period for taking any subsequent action in connection with this appeal may be extended under 37 C.F.R. § 1.136(a)(1)(iv).

## AFFIRMED

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